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ABSTRACT

Values of yield strength, tensile strength, elongation, and Young's Modulus have been determined on 0.015" diameter zirconium wire at temperatures ranging from 70° F. to 1000° F.

INTRODUCTION:

In view of the fact that possible applications of zirconium metal involve its use at elevated temperatures, a knowledge of its elevated temperature mechanical properties will be of value. Although the mechanical properties of zirconium have been investigated rather thoroughly at room temperature, a relatively small amount of work has been done at elevated temperatures. Boulger[1] summarizes the available short time tensile properties of zirconium. These are given in Table I.

TABLE I

Material	Atmosphere	Test Temp., °C	Elong. %	Red. in Area, %	Tensile Strength, psi
Iodide (extruded)	air	20	23.7	39.7	65,000
" "	argon	250	32.7	55.1	40,000
" "	argon	500	39.7	68.0	28,500
" "	argon	750	99.6	97.8	8,000
Iodide Crystal Bar	helium	500			24,000
	nitrogen	500			15,000
	helium	700			10,000
	nitrogen	700			7,000
	helium	800			6,600
	nitrogen	800			500

Elevated temperature hardness values for zirconium produced by reduction of the chloride by magnesium have been reported by Hayes, et al[2]. Values of Brinell hardness taken from a plot of hardness versus temperature are as follows:

TABLE II

Test Temp., °C	Brinell Hardness of	
	Cast Zr	Rolled Zr
20	120	200
100	95	180
200	-	125
250	55	110
300	45	95
400	40	75
500	35	50
600	25	30

Boulger[1] gives the following data on the room temperature tensile properties of zirconium:

TABLE III

	A	B	C
Yield strength (0.2% offset) (psi)	15,000	69,000	14,500
Ultimate strength (psi)	34,500	86,000	39,500
Reduction in area, %	26	34	35
Elongation, % (four diameter gage length)	28	17	33
Rockwell hardness	B-25 F-70	B-87 F-105	B-42 F-83

Note: A - 0.25" diameter rod machined from as-deposited bars

B - 0.125" diameter rod, reduced from above by cold swaging 65%

C - Same as B, annealed at 705° C. in vacuo

Material C, which has been cold worked and annealed has higher tensile strength, as well as higher elongation and reduction in area, than has the material in the as-deposited condition. This effect is usually noted in material worked down to small sizes.

Table IV shows the effect of annealing temperature on tensile strength and elongation; very marked changes in the curves of tensile strength and elongation versus tempering temperature occur between 400° C. and 500° C., indicating that the recrystallization temperature lies in this range.

TABLE IV

	Elong. %	Ultimate Strength, psi
0.02" wire, cold swaged	3	115,000
" " 3 hrs., 400° C.	2-1/2	101,000
" " 3 " 500° C.	12	56,000
" " 1 hr., 600° C.	12-1/2	51,000
" " 1 " 700° C.	12-1/2	44,000
" " 1 " 800° C.	11	38,500
" " 1 " 900° C.	15-1/2	43,000
" " 1/4 " 1000° C.	15	44,000
0.02" wire*, hard drawn	2	128,800
" " tempered 455° C.	5-1/2	93,500
" " annealed 790° C.	14	61,000

Note: Elongation values for 5.9" gage length.
All materials processed from iodide crystal bar.

* Different investigation.

The potent hardening effect of small amounts of impurities (probably mainly oxygen and nitrogen) is shown in Table V. The tensile strength of magnesium-reduced zirconium is about 75% higher than that of iodide zirconium; this is probably due to the greater amount of impurities present in the magnesium reduced material.

TABLE V

Material	Processing Treatment	Red. in Area, %	Elong. %	Yield Strength	Ultimate Strength
Iodide Zr (Rockwell B-38)	As rec'd	40.0	13.6	18,750	40,750
	Vacuum melted in graphite, cast, extruded at 800° C.	39.7	23.7	-	64,750
	As above, extruded at 1060° C.	46.2	21.5	35,250	56,500
Mg-reduced Zr Rockwell B-84	As rec'd	31.4	13.7	43,500	68,500
	As rec'd metal canned, extruded at 800° C.	43.8	15.3	42,750	80,250
	As above, extruded at 1060° C.	43.7	19.9	47,750	79,500

EXPERIMENTAL:

A sketch of the furnace designed for these tests is shown in Fig. 1. The furnace proper consisted of a one inch diameter stainless steel tube having an insulating layer of asbestos paper soaked in water glass on the outer surface. On this was wound a nichrome heating element. Over this winding was applied a coating of alundum cement. Rather than attempting to remove temperature gradients by taper winding the furnace, the required constant temperature was obtained by applying varying thicknesses of alundum cement. By this method, a central zone six inches long was obtained which varied $\pm$ 2° F. The protective atmosphere, in this case argon, was preheated to the testing temperature by passing it through the gas heating furnace shown in the lower right of the figure.

The tank argon used as protective atmosphere was purified by passing it through two drying towers filled with magnesium perchlorate and over uranium chips maintained at 650° - 700° C.

The testing furnace was gas-tight at all points except where the upper grip passed loosely through the top of the furnace. The argon was exhausted under positive pressure through this annular hole at a flow rate of approximately 3 to 5 cubic feet per hour, preventing entrance of air. The specimens were slightly darkened after testing, indicating that a small amount of oxidation occurred probably just after the furnace was closed after mounting a new specimen.

The material tested was 0.015 inch diameter zirconium wire produced from iodide crystal bar by Foote Mineral Co. (Lot No. 910-65). Its processing history was as follows:

TABLE VI

1. Crystal bar cold rolled to 0.125 inch
2. Vacuum annealed at 1250° F. for 1 minute
3. Cold swaged through 0.125 inch die
4. Sheathed in brass
5. Swaged to 0.086 inch
6. Sheathed wire drawn to 0.0253 inch
7. Brass sheath removed in HNO_3
8. Vacuum annealed on spools at 1300° F. for 45 minutes.

Room temperature tensile properties of this material are (average of six values):

Yield Strength	-	22,000 psi
Tensile Strength	-	45,000 "
Elongation (4" gage)	-	18%

These are about the values to be expected from a study of the data in Table IV, showing 0.02 inch wire annealed at 700° C. (our material annealed at 704° C.) to have a tensile strength of 44,000 psi and an elongation in 5.9" gage length of 12-1/2%. The material used in this investigation, therefore, appears to be about fully annealed. It is known that this material represents part of a low hafnium content lot.

The tensile testing machine used in this investigation was a Tinius Olsen model developed for Sylvania Electric Products for use in testing wire. Its low load ranges (1000 and 2500 grams full scale) and high elongation magnification (10 to 20 times) were designed for precise measurements.

Special arrangements had to be made to grip this small diameter wire, inasmuch as the standard wire grips were too large to fit inside the furnace. This difficulty was overcome by using collets of one inch lengths of copper tubing approximately 0.080" O.D. and 0.020" I.D.; these were crimped on the ends of the specimens and in turn were clamped into a 3/32" diameter hole in the grips by means of set screws.

Four inch gage length specimens were used for determination of tensile strength, yield strength (0.2% offset), and elongation, while five inch gage length specimens were used for the determination of Young's Modulus. Testing procedure was as follows: The furnace (at the desired temperature) was raised so that the grips were exposed. The specimen was clamped in the grips as quickly as possible, and the furnace was lowered so that the taper joints formed an air tight seal. (These joints were lubricated with a silicone stop-cock grease which remained quite viscous throughout the tests). When the temperature, as read on the chromel-alumel thermocouple near the center of the test specimen, showed the temperature to be constant at the desired level for five minutes, the test was run. Tests made to determine tensile strength, yield strength, and elongation were made at head rates of 0.2, 0.5, and 2.0 inches per minute, corresponding to strain rates on four inch specimens of 0.05, 0.125, and 0.50 inch/inch/minute. Tests made to determine Young's Modulus were made at a head rate of 0.025 inch per minute, corresponding to a strain rate on a five inch specimen of 0.005 inch/inch/minute. The tests for this determination were made in much the same manner as the others. However, the specimens were not run to fracture, and four determinations were made on each specimen.

DISCUSSION OF RESULTS:

The results of our tests are tabulated below:

TABLE VII

Test Temp. °F	Tensile Strength psi	Yield Strength psi	Elongation % in 4"
	STRAIN RATE = 0.05 INCH/INCH/MINUTE		
70	45100 45100	20700 20700	19.3 18.0
200	40800 41400	21900 21300	16.8 16.3
400	32400 33200	18300 18800	15.3 15.0
600	25700 25800	15200 15400	14.3 13.6
800	19100 18600	13500 13600	13.6 13.9
1000	10100 10500	9400 9000	32.8 44.5
	STRAIN RATE = 0.125 INCH/INCH/MINUTE		
70	45500 45400	22500 24400	19.9 18.3
200	38800 40600	20400 21700	17.9 18.3
400	33500 33600	18900 19000	16.5 16.5
600	26800 26200	15800 15500	14.0 14.5
800	20400 20200	14000 14500	14.3 15.8
1000	11600 11500	9600 10700	28.8 24.8

TABLE VII (cont'd)

Test Temp. °F	STRAIN RATE = 0.50 INCH/INCH/MINUTE		
	Tensile Strength psi	Yield Strength psi	Elongation % in 4"
70	46300 46300	23800 20100	17.8 17.3
200	41400 42000	21900 23800	17.3 17.8
400	34000 34100	19800 19700	15.5 15.8
600	27200 28200	19600 20600	12.4 14.9
800	20400 20800	15400 15800	12.4 13.1
1000	14000 14100	11300 11600	23.3 22.3

TABLE VIII

STRAIN RATE = 0.005 INCH/INCH/MINUTE

Test Temp. °F	Young's Modulus psi
70	9.3×10^6
200	7.9×10^6
400	5.9×10^6
600	5.6×10^6
800	5.6×10^6
1000	4.2×10^6

These data are shown graphically in Figures 2 through 4, plotting tensile strength and yield strength (Fig. 2), elongation (Fig. 3), and Young's Modulus (Fig. 4) versus temperature. Both tensile strength and yield strength decrease with increasing temperature, with a more or less linear relationship appearing between the strength value and temperature. It is interesting to note in Fig. 2 that the tensile strength decreases much more rapidly with increasing temperature than does yield strength, until at 1000° F. the values are almost the same. The lack of strain hardening, that is, little or no difference between yield and tensile strengths, indicates that the material is recrystallizing at the same time that it is being strained. It should also be noted that increasing the strain rate appears to increase the strength values, although the effect is not marked in the ranges of strain rates investigated.

Elongation may be seen to decrease with increasing temperature, at least up to the range 800° - 1000° F. There the elongation increased very rapidly with increasing temperature; the variation in elongation values at 1000° F. indicates that the material is in a somewhat unstable condition, which may be associated with the recrystallization phenomenon discussed in the previous paragraph.

The graph of Young's Modulus versus temperature again shows a decrease with increasing temperature. Our value for Young's Modulus at room temperature (9.3×10^6 psi) is somewhat lower than previously reported values, which range from 11 to 14×10^6 psi. Battelle has reported a linear decrease in Young's Modulus from 13.2×10^6 psi at 70° F. to 10.6×10^6 psi at 600° F.

CONCLUSIONS:

Tensile tests on 0.015 inch diameter annealed zirconium wire in the temperature range 70° - 1000° F. show that:

1. Tensile strength decreases linearly from approximately 45,000 psi at 70° F. to 10,000 psi at 1000° F.

2. Yield strength decreases from about 20,000 psi at 70° F. to 9000 psi at 1000° F.

3. Elongation decreases slightly from its room temperature value of 18% as temperature increases. In the range 800° - 1000° F, the elongation increases to about 30%.

4. Young's Modulus decreases in a curvilinear manner from 9.3×10^6 at 70° F. to 4.2×10^6 at 1000° F.

The effect of degree of preferred orientation on Young's Modulus is being studied at present; a metallographic study of recrystallization during testing at 1000° F. is also being made. Results will be reported as they are available.

REFERENCES

1. F. Boulger, "The Properties of Zirconium", BMI T-10, March 15, 1949.

2. E.T. Hayes, E.D. Dilling and A.H. Roberson, "Fabrication and Mechanical Properties of Ductile Zirconium", American Society for Metals preprint No. 32, 1949.

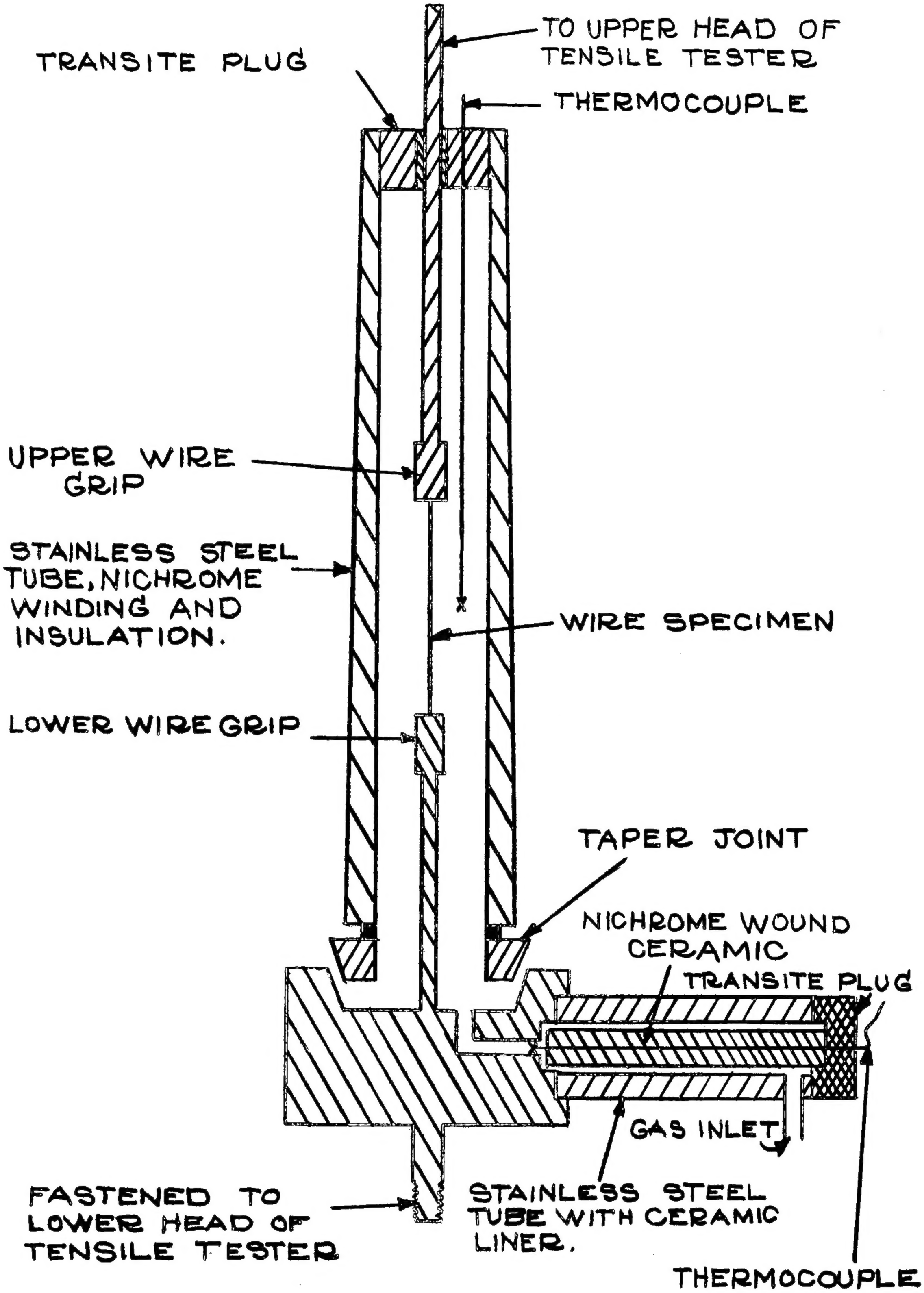

TO UPPER HEAD OF TENSILE TESTER
TRANSITE PLUG
THERMOCOUPLE
UPPER WIRE GRIP
STAINLESS STEEL TUBE, NICHROME WINDING AND INSULATION.
WIRE SPECIMEN
LOWER WIRE GRIP
TAPER JOINT
NICHROME WOUND CERAMIC
TRANSITE PLUG
GAS INLET
FASTENED TO LOWER HEAD OF TENSILE TESTER
STAINLESS STEEL TUBE WITH CERAMIC LINER.
THERMOCOUPLE

FIG-1.

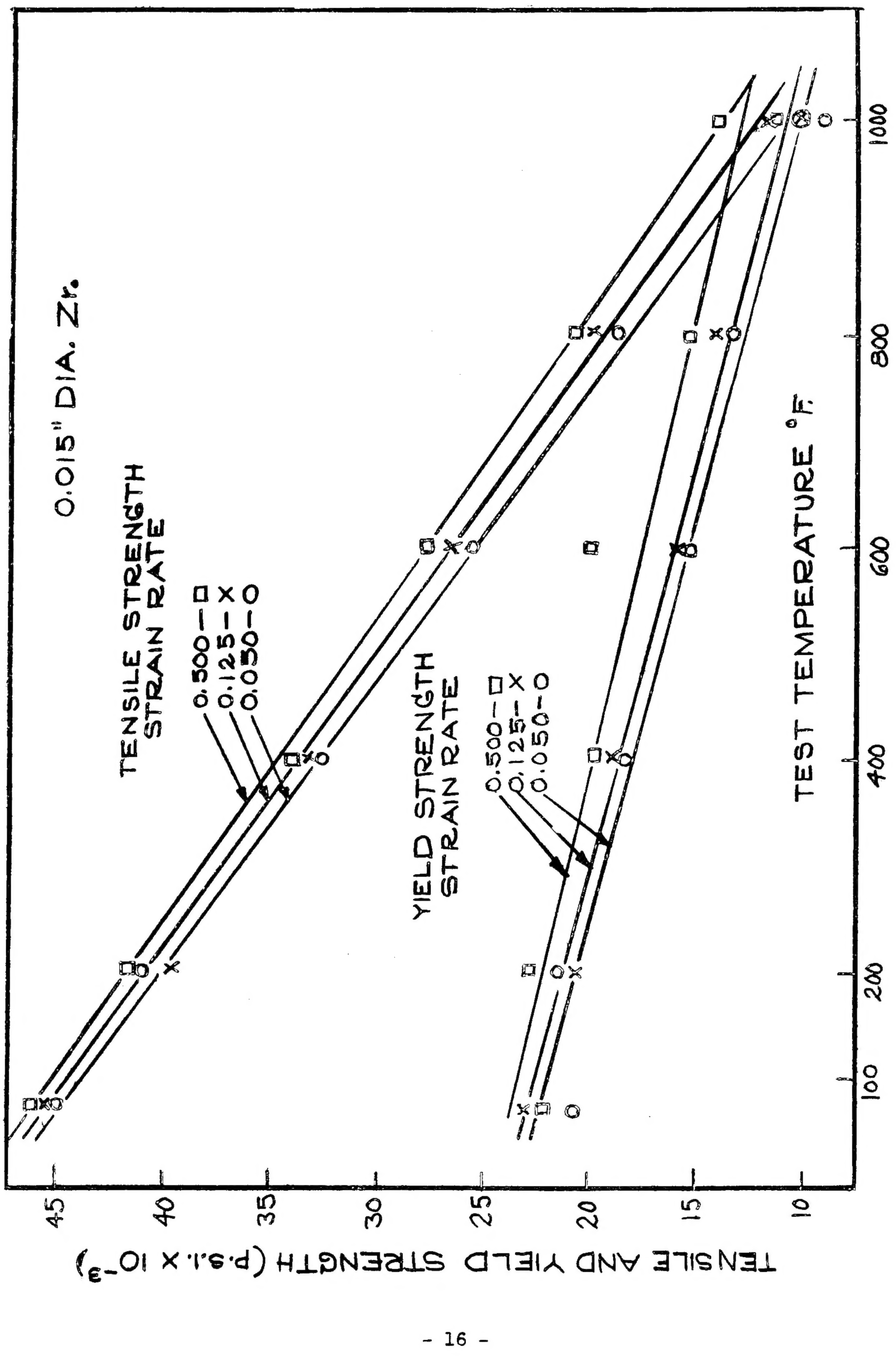
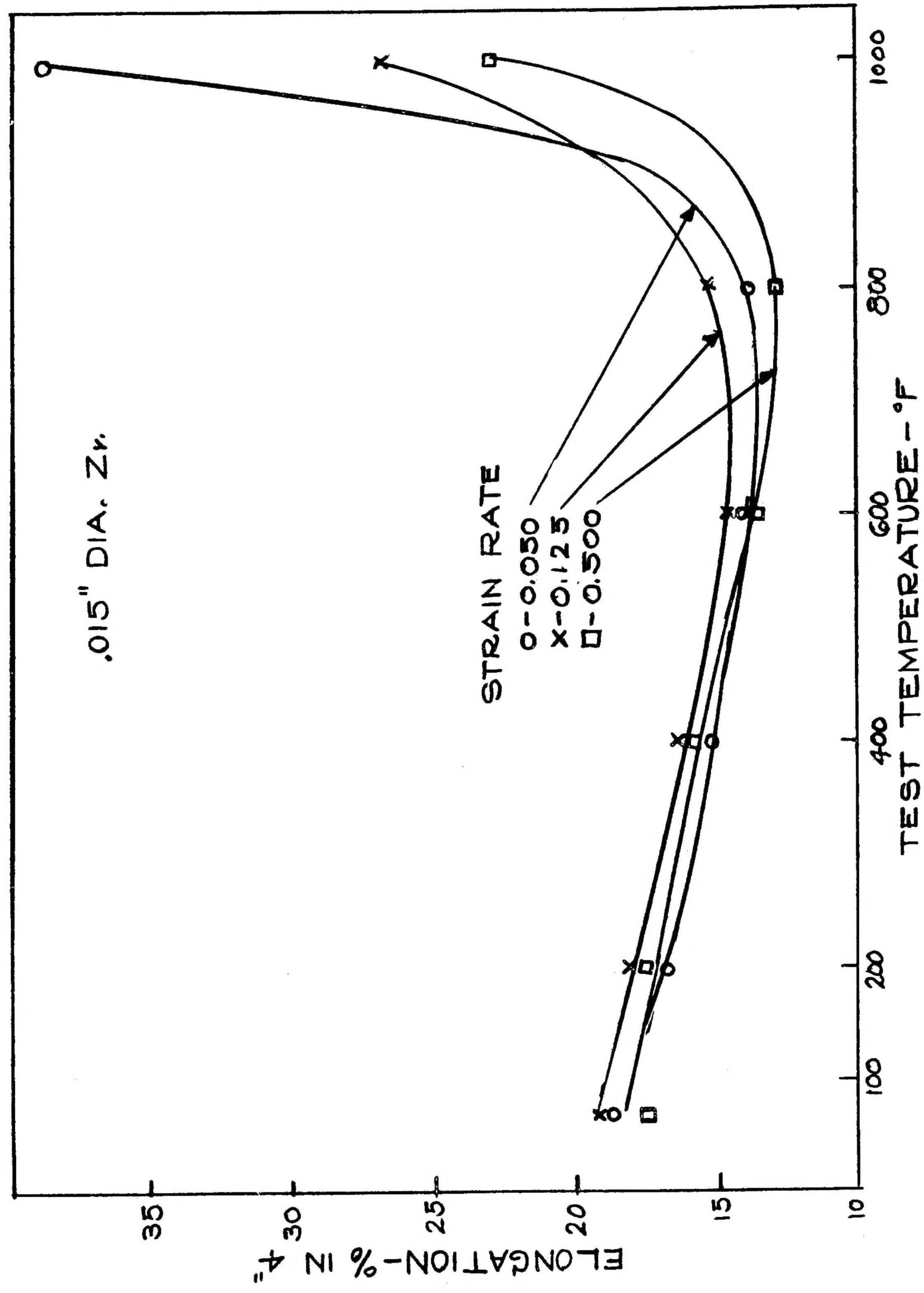

0.015" DIA. Zr.
TENSILE STRENGTH
STRAIN RATE
0.500 — □
0.125 — X
0.050 — O
YIELD STRENGTH
STRAIN RATE
0.500 — □
0.125 — X
0.050 — O
TEST TEMPERATURE °F
100
200
400
600
800
1000
TENSILE AND YIELD STRENGTH (p.s.i. X 10^{-3})
45
40
35
30
25
20
15
10

FIG-2


.015" DIA. Zr.
STRAIN RATE
O - 0.050
X - 0.125
□ - 0.500
ELONGATION - % IN 4"
10
15
20
25
30
35
100
200
400
600
800
1000
TEST TEMPERATURE - °F
FIG. 3

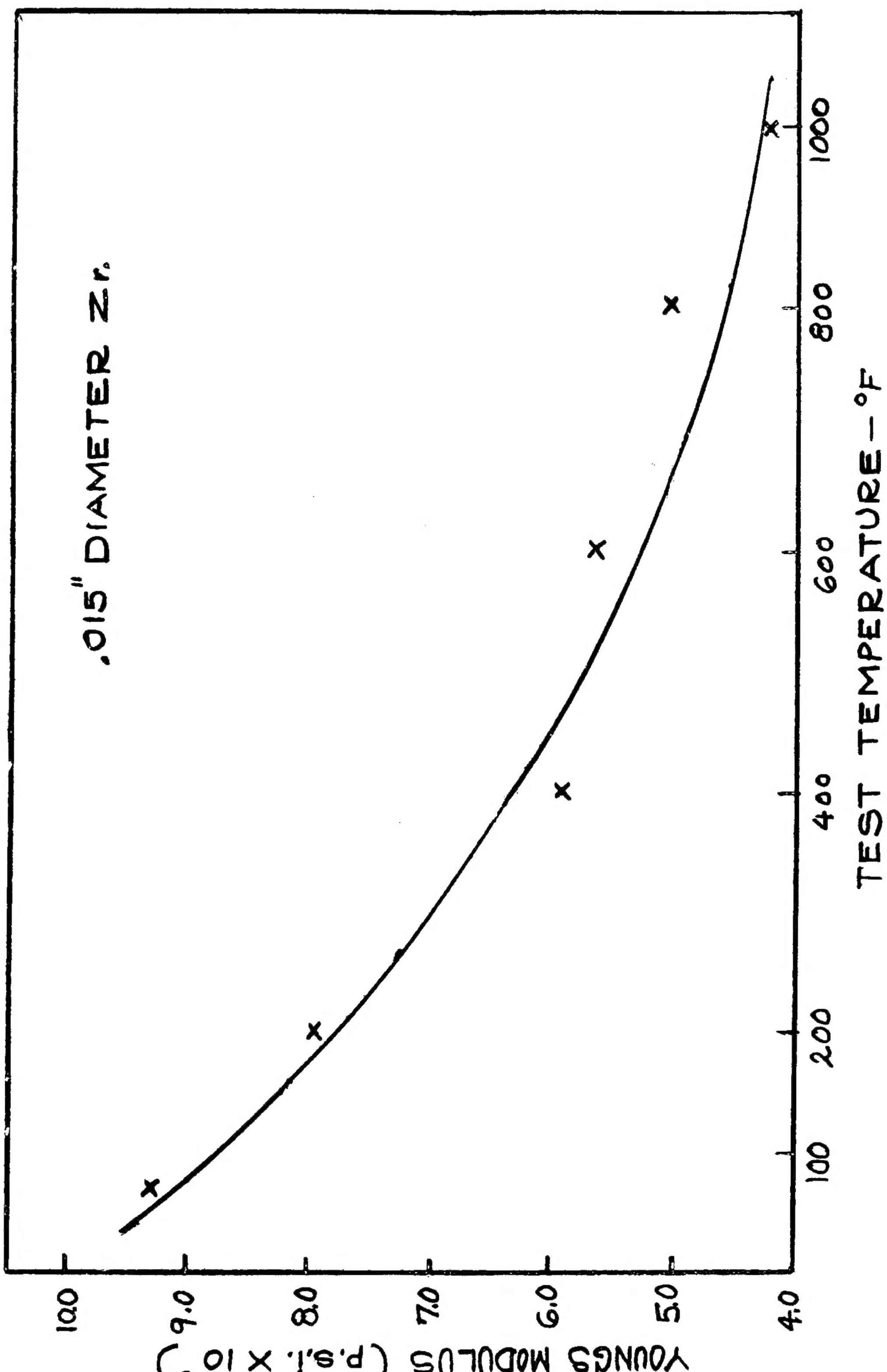

.015" DIAMETER Zr.
YOUNGS MODULUS (p.s.i. X 10^{-6})
10.0
9.0
8.0
7.0
6.0
5.0
4.0
100
200
400
600
800
1000
TEST TEMPERATURE — °F

FIG. 4